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(54) Title: HERBICIDAL o-CARBOMETHOXYLSULFONYLUREAS			
<p>(I)</p>			
(57) Abstract <p>The compound of formula (I) is useful as an agricultural chemical. It has high herbicidal activity, desirable spectrum of weed control and low residual activity, as such, it is particularly useful for fallow.</p>			

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TITLEHERBICIDAL o-CARBOMETHOXYLSULFONYLUREASRelated Application

This application is a continuation-in-part of U.S. Serial No. 101,703, filed on September 28, 1987.

Background of the Invention

A designated o-carbomethoxysulfonylurea is useful as an agricultural chemical. It has high
10 herbicidal activity, a desirable spectrum of weed control and low residual activity, as such, it is particularly useful for fallow.

The compound of interest is disclosed in U.S. Patent 4,435,206, issued 3/6/84 and in EP-A-184,385,
15 published 6/11/86, but not for all utilities.

The importance of cereal crops, such as wheat and barley, and plantation crops for feeding mankind is well known. Unfortunately, some of the known herbicides have high residual activity or are unsafe
20 to crops. Thus, the crops cannot be easily rotated in an area in which known herbicides are present.

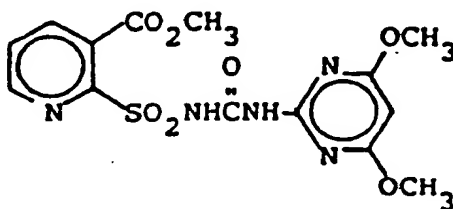
The present invention helps fill the need for herbicides with high herbicidal activity and low residual activity.

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Summary of the Invention

This invention pertains to the use of the compound of Formula I, its agriculturally suitable salts for broad spectrum weed control in fallow land
30 and/or its use to control undesired vegetation in turf and plantation crops.

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I

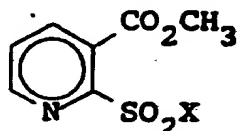
5 The compound is 2-[[[(4,6-dimethoxypyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-3-pyridinecarboxylic acid, methyl ester.

The compound also undergoes rapid soil dissipation and would ordinarily possess no recrop limitations.

Detailed Description of the Invention

15 As part of the present invention, it has been found that unexpectedly high herbicidal activity with a desirable period of control for undesired vegetation in noncrop areas and fallow, as well as safety to turf and plantation crops, is exhibited by 2-[[[(4,6-dimethoxypyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-3-pyridinecarboxylic acid, methyl ester.

20 The following intermediates of Formula II are considered to be novel.



25 **II**

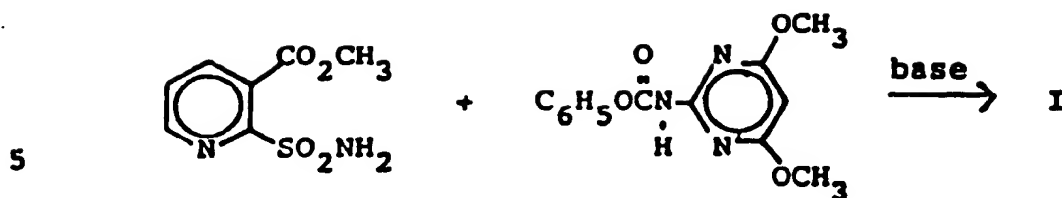
wherein

X is Cl, NH_2 , $\text{NHC}(\text{CH}_3)_3$ and $\text{HNC}(\text{O})\text{OC}_6\text{H}_5$.

Synthesis

30 The title compound of Formula I can be prepared by the reaction of 2-(aminosulfonyl)-3-pyridinecarboxylic acid, methyl ester and 4,6-dimethoxy-2-pyrimidinyl carbamic acid, phenyl ester in an inert aprotic solvent, such as toluene, acetonitrile, tetrahydrofuran, dichloromethane or dioxane, and in
35 the presence of a base, such as 1,8-diazabicyclo-[5.4.0]undec-7-ene (DBU) or N,N-dimethylaniline.

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The following examples further illustrate the present invention.

10

Example 1

2-(Phenylmethylthio)-3-pyridinecarboxylic acid chloride

To a stirred suspension of 300 g of 2-(phenylmethylthio)-3-pyridinecarboxylic acid in benzene was added 268 mL of thionyl chloride, dropwise. The resultant yellow suspension was heated to reflux for 4 hours, allowed to cool to room temperature overnight and then concentrated under reduced pressure. The solid residue was washed with cold hexanes and dried in vacuo at room temperature to give 309.3 g of a tan powder, m.p. 94-96°C; IR 1730 cm^{-1} (C(O)); NMR (90 MHz, CDCl_3) δ 8.75 (dd, 2H, ArH), 7.5 (m, 6H, ArH), 4.5 (s, 2H, CH_2).

25

Example 2

2-(Phenylmethylthio)-3-pyridinecarboxylic acid, methyl ester

To a stirred solution of 4 L of methanol under a nitrogen atmosphere was added 450 g of 2-(phenylmethylthio)-3-pyridinecarboxylic acid chloride, followed by the dropwise addition of 382 mL of triethylamine. The resultant mixture was heated to reflux for two hours, then allowed to stir at room temperature for 4 days. The reaction mixture was added to a battery jar containing 8 L of ice water. The resultant suspension

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was filtered through a sintered glass funnel and the residue was air dried and later dried in a vacuum oven at room temperature, to give 418.5 g of the desired product as a tan powder, m.p. 37-38°C; IR 1715 cm⁻¹ (C(O)); NMR (90MHz, CDCl₃) δ 8.75 (dd, 1H, ArH), 8.3 (dd, 1H, ArH), 7.5 (m, 6H, ArH), 4.45 (s, 2H, CH₂) and 3.9 (s, 3H, CH₃).

103

Example 32-[(1,1-Dimethylethyl)aminosulfonyl]-3-pyridinecarboxylic acid, methyl ester

A mixture of 100 g of 2-(phenylmethylthio)-3-pyridinecarboxylic acid, methyl ester, 500 mL of water and 500 mL of Freon® TF was cooled to 0°C, and 70.9 mL of chlorine was then added via a gas dispersion tube. The resultant suspension was allowed to stir at 5°C for 1 hour. The precipitate was collected and washed with cold Freon® TF, to give a white powder, m.p. 48-50°C. This white solid was added to 1 L of dichloromethane at -50°C under a nitrogen atmosphere. To this solution, cooled to -60°C, was added dropwise, 226 g of t-butylamine. After warming to room temperature and dilution with 2 L of water, the organic layer was separated and the aqueous layer was extracted twice with 1 L of dichloromethane. The combined organic layers were washed with brine, dried over magnesium sulfate, filtered and the filtrate was concentrated under reduced pressure. The resultant yellow solid was slurried in cold n-butyl chloride, filtered and the solid collected to give 80.25 g of a white powder m.p. 160-161°C; IR (nujol) 3160 cm⁻¹ (NH) and 1745 cm⁻¹ (C(O)); NMR (90 MHz, CDCl₃) δ 9.0 (dd, 1H, ArH), 8.3 (dd, 1H, ArH); 7.8 (m, 1H, ArH), 5.8 (bs, 1H, NH), 4.1 (s, 3H, CH₃) and 1.35 (s, 9H, CH₃).

Example 42-(Aminosulfonyl)-3-pyridinecarboxylic acid, methyl ester

To 930 g of trifluoroacetic acid was added portionwise 50 g of 2-[(1,1-dimethylethyl)aminosulfonyl]-3-pyridinecarboxylic acid, methyl ester. The mixture was allowed to stir at room temperature for 3 days and then concentrated under reduced pressure. After addition of 150 mL of cold diethyl ether the resultant solid was collected. This solid was washed with cold ether and air dried to give 37.6 g of a white powder, m.p. 147-149°C; IR (nujol) 3360 cm^{-1} (NH_2), 1720 cm^{-1} (C(O)) and 1070 cm^{-1} (SO_2); NMR (DMSO-d_6) δ 9.0 (dd, 1H, ArH), 8.3 (dd, 1H, ArH), 7.9 (q, 1H, ArH), 7.2 (bs, 2H, NH_2) and 3.9 (s, 3H, CH_3).

Example 52-[[[4,6-Dimethoxypyrimidin-2-yl]aminocarbonyl]lamino-sulfonyl]-3-pyridinecarboxylic acid, methyl ester

To a mixture of 346.4 g of 2-(aminosulfonyl)-3-pyridinecarboxylic acid, methyl ester and 485 g of 4,6-dimethoxy-2-pyrimidinyl carbamic acid, phenyl ester in 3,464 mL of dry acetonitrile was added dropwise 303 mL of DBU. After stirring the solution for 2 hours at room temperature, 3 teaspoons of activated carbon were added. The mixture was then filtered through Celite® to give a clear solution. The filtrate was diluted with 3.5 L of ice water and slowly concentrated hydrogen chloride was added until a pH of 3.5 was obtained. The solid was collected, washed with water, then diethyl ether and air dried to give 539.2 g of the desired product as a white powder, m.p. 158-161°C; IR (nujol) 1735, 1745 cm^{-1} (2 X C(O)), 1510 cm^{-1} (NH) and 1080 cm^{-1} (SO_2); NMR (90 MHz, CDCl_3) δ 11.8 (bs, 1H, NH), 8.9 (dd, 1H,

ArH), 8.3⁻ (dd, 1H, ArH), 7.75 (q, 1H, ArH), 7.6 (bs, 1H, NH), 5.9 (s, ArH, 1H) and 4.1 (s, 9H, CH₃).

5 Formulations

Useful formulations of the compound of Formula I can be prepared in conventional ways. They include dusts, granules, pellets, solutions, suspensions, emulsions, wettable powders, emulsifiable concentrates and the like. Many of these may be applied directly. Sprayable formulations can be extended in suitable media and used at spray volumes of from a few liters to several hundred liters per hectare. High strength compositions are primarily used as intermediates for further formulation. The formulations, broadly, contain about 0.1% to 99% by weight of active ingredient(s) and at least one of (a) about 0.1% to 20% surfactant(s) and (b) about 1% to 99.9% solid or liquid diluent(s). More specifically, they will contain these ingredients in the following approximate proportions:

		Weight Percent*		
		Active Ingredient	Diluent(s)	Surfactant(s)
25	Wettable Powders	20-90	0-74	1-10
	Oil Suspensions, Emulsions, Solutions, (including Emulsifi- able Concentrates)	3-50	40-95	0-15
30	Aqueous Suspension	10-50	40-84	1-20
	Dusts	1-25	70-99	0-5
	Granules and Pellets	0.1-95	5-99.9	0-15
35	High Strength Compositions	90-99	0-10	0-2

- * Active ingredient plus at least one of a
Surfactant or a Diluent equals 100 weight percent.

5

Lower or higher levels of active ingredient can, of course, be present depending on the intended use and the physical properties of the compound. Higher ratios of surfactant to active ingredient are some-- times desirable, and are achieved by
10: incorporation into the formulation or by tank mixing.

Typical solid diluents are described in Watkins, et al., "Handbook of Insecticide Dust Diluents and Carriers", 2nd Ed., Dorland Books,
15: Caldwell, New Jersey, but other solids, either mined or manufactured, may be used. The more absorptive diluents are preferred for wettable powders and the denser ones for dusts. Typical liquid diluents and solvents are described in Marsden, "Solvents Guide,"
20: 2nd Ed., Interscience, New York, 1950. Solubility under 0.1% is preferred for suspension concentrates; solution concentrates are preferably stable against phase separation at 0°C. "McCutcheon's Detergents and Emulsifiers Annual," MC Publishing Corp.,
25: Ridgewood, New Jersey, as well as Sisely and Wood, "Encyclopedia of Surface Active Agents," Chemical Publishing Co., Inc., New York, 1964, list surfactants and recommended uses. All formulations can contain minor amounts of additives to reduce
30: foaming, caking, corrosion, microbiological growth, and the like.

The methods of making such compositions are well-known. Solutions are prepared by simply mixing the ingredients. Fine solid compositions are made by
35: blending and, usually, grinding as in a hammer or

fluid energy mill. Suspensions are prepared by wet milling (see, for example, Littler, U.S. Patent 3,060,084). Granules and pellets may be made by
5 spraying the active material upon preformed granular carriers or by agglomeration techniques. See Browning, "Agglomeration", Chemical Engineering, December 4, 1967, pp. 147ff. and "Perry's Chemical Engineer's Handbook", 5th Ed., McGraw-Hill, New York,
10 1973, pages 8 to 57 and following.

For further information regarding the art of formulation, see for example: U.S. Patent 3,235,361, column 6, line 16 through column 7, line 19 and Examples 10 through 41; U.S. Patent 3,309,192, column 5,
15 line 43 through column 7, line 62 and Examples 8, 12, 15, 39, 41, 52, 53, 58, 132, 138 to 140, 162 to 164, 166, 167 and 169 to 182; U.S. Patent 2,891,855, column 3, line 66 through column 5, line 17 and Examples 1 to 4; Klingman, "Weed Control as a
20 Science", John Wiley and Sons, Inc., New York, 1961, pages 81 to 96; and Fryer et al., "Weed Control Handbook", 5th Ed., Blackwell Scientific Publications, Oxford, 1968, pages 101 to 103.

In the following Examples, all parts are by
25 weight unless otherwise indicated.

Example 6

Wettable Powder

2-[[[(4,6-dimethoxypyrimidin-2-yl)aminocarbonyl]amino-
30 sulfonyl]-3-pyridinecarboxylic acid, methyl ester 50%
sodium alkylnaphthalenesulfonate 2%
low viscosity methyl cellulose 2%
diatomaceous earth 46%

The ingredients are blended, coarsely hammer-milled and then air-milled to produce particles essentially all below 10 microns in diameter. The product is reblended before packaging.

Example 7

Granule

	Wettable Powder of Example 6	5%
	attapulgit granules	95%
10	(U.S.S. 20 to 40 mesh; 0.84 to 0.42 mm)	

A slurry of wettable powder containing 25% solids is sprayed on the surface of attapulgit granules in a double-cone blender. The granules are dried and packaged.

15

Example 8

Extruded Pellet

	2-[[[(4,6-dimethoxypyrimidin-2-yl)aminocarbonyl]amino-sulfonyl]-3-pyridinecarboxylic acid, methyl ester	25%
20	anhydrous sodium sulfate	10%
	crude calcium ligninsulfonate	5%
	sodium alkyl naphthalenesulfonate	1%
	calcium/magnesium bentonite	59%

The ingredients are blended, hammer-milled and then moistened with about 12% water. The mixture is extruded as cylinders about 3 mm diameter which are cut to produce pellets about 3 mm long. These may be used directly after drying, or the dried pellets may be crushed to pass a U.S.S. No. 20 sieve (0.84 mm openings). The granules held on a U.S.S. No. 40 sieve (0.42 mm openings) may be packaged for use and the fines recycled.

35

Example 9

5

Low Strength Granule

2-[[[(4,6-dimethoxypyrimidin-2-yl)aminocarbonyl]amino-
sulfonyl]-3-pyridinecarboxylic acid, methyl ester 1%
N,N-dimethylformamide 9%
attapulgit granules 90%

10

(U.S.S. 20 to 40 sieve, 0.42 to 0.84 mm)

The active ingredient is dissolved in the solvent
and the solution is sprayed upon dedusted granules in
a double cone blender. After spraying of the solution
has been completed, the blender is allowed to run for
a short period and then the granules are packaged.

15

Example 10Aqueous Suspension

20

2-[[[(4,6-dimethoxypyrimidin-2-yl)aminocarbonyl]amino-
sulfonyl]-3-pyridinecarboxylic acid, methyl ester 40%
polyacrylic acid thickener 0.3%
dodécylphenol polyethylene glycol ether 0.5%
disodium phosphate 1%
monosodium phosphate 0.5%
polyvinyl alcohol 1.0%
water 56.7%

25

The ingredients are blended and ground together in
a sand mill to produce particles essentially all under 5
microns in size.

30

Example 11Oil Suspension

2-[[[(4,6-dimethoxypyrimidin-2-yl)aminocarbonyl]amino-
sulfonyl]-3-pyridinecarboxylic acid, methyl ester 35%

35

blend of polyalcohol carboxylic 6%
esters and oil soluble petroleum
sulfonates
xylene 59%

The ingredients are combined and ground together in a sand mill to produce particles essentially all below 5 microns. The product can be used directly, extended with oils, or emulsified in water.

Example 12

Granule

2-[[[(4,6-dimethoxypyrimidin-2-yl)aminocarbonyl]amino-
sulfonyl]-3-pyridinecarboxylic acid, methyl ester 80%
wetting agent 1%
crude ligninsulfonate salt (containing 10%
5 to 20% of the natural sugars)
attapulgitic clay 9%

The ingredients are blended and milled to pass through a 100 mesh (0.149 mm opening) screen. This material is then added to a fluid bed granulator, the air flow is adjusted to gently fluidize the material, and a fine spray of water is sprayed onto the fluidized material. The fluidization and spraying are continued until granules of the desired size range are made. The spraying is stopped, but fluidization is continued, optionally with heat, until the water content is reduced to the desired level, generally less than 1%. The material is then discharged, screened to the desired size range, generally 14 to 100 mesh (1410 to 149 microns), and packaged for use.

Example 13

High Strength Concentrate

2-[[[(4,6-dimethoxypyrimidin-2-yl)aminocarbonyl]amino-
sulfonyl]-3-pyridinecarboxylic acid, methyl ester 99%

12

silica a rog 1	0.5%
synthetic amorphous silica	0.5%

The ingredients are blended and ground in a hammer-mill to produce a material essentially all passing a U.S.S. No. 50 screen (0.3 mm opening). The concentrate may be formulated further if necessary.

Example 1410 Wettable Powder

2-[[[(4,6-dimethoxypyrimidin-2-yl)aminocarbonyl]amino-sulfonyl]-3-pyridinecarboxylic acid, methyl ester	90%
dioctyl sodium sulfosuccinate	0.1%
synthetic fine silica	9.9%

15 The ingredients are blended and ground in a hammer-mill to produce particles essentially all below 100 microns. The material is sifted through a U.S.S. No. 50 screen and then packaged.

20

Example 15Wettable Powder

2-[[[(4,6-dimethoxypyrimidin-2-yl)aminocarbonyl]amino-sulfonyl]-3-pyridinecarboxylic acid, methyl ester	20%
sodium ligninsulfonate	20%
montmorillonite clay	60%

The ingredients are thoroughly blended, coarsely hammer-milled and then air-milled to produce particles essentially all below 10 microns in size. The material is reblended and then packaged.

30

Example 16Dust

2-[[[(4,6-dimethoxypyrimidin-2-yl)aminocarbonyl]amino-sulfonyl]-3-pyridinecarboxylic acid, methyl ester	10%
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35

attapulgit 10%
Pyrophyllite 80%

The active ingredient is blended with attapul-
gite and then passed through a hammer-mill to produce
particles substantially all below 200 microns. The
ground concentrate is then blended with powdered
pyrophyllite until homogeneous.

The compound of this invention may be used in
combination with other commercial herbicides, plant
growth regulants, insecticides or fungicides. Weed
Research, Volume 26 (1986) pages 441 to 445, the
disclosure of which is incorporated herein by reference,
specifically names 294 commonly used herbicides and
plant growth regulants. The following list exemplifies
some of the herbicides suitable for use in mixtures.

	<u>Common Name</u>	<u>Chemical Name</u>
	acetochlor	2-chloro-N-(ethoxymethyl)-N-(2-ethyl-6-methylphenyl)acetamide
5	acifluorfen	5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitrobenzoic acid
	acrolein	2-propenal
	alachlor	2-chloro-N-(2,6-diethylphenyl)-N-(methoxymethyl)acetamide
10	ametryn	N-ethyl-N'-(1-methylethyl)-6-(methylthio)-1,3,5-triazine-2,4-diamine
	amitrole	1H-1,2,4-triazol-3-amine
	AMS	ammonium sulfamate
15	asulam	methyl [(4-aminophenyl)sulfonyl]carbamate
	atrazine	6-chloro-N-ethyl-N'-(1-methylethyl)-1,3,5-triazine-2,4-diamine
	barban	4-chloro-2-butynyl 3-chlorocarbamate
20	benefin	N-butyl-N-ethyl-2,6-dinitro-4-(trifluoromethyl)benzenamine
	bensulfuron methyl	2-[[[[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonyl]amino]sulfonyl]methyl]benzoic acid, methyl ester
25	bensulide	O,O-bis(1-methylethyl) S-[2-[(phenylsulfonyl)amino]ethyl] phosphorodithioate
	bentazon	3-(1-methylethyl)-(1H)-2,1,3-benzothiadiazin-4(3H)-one, 2,2-dioxide
	benzofluor	N-[4-(ethylthio)-2-(trifluoromethyl)-phenyl]methanesulfonamide
30	benzoylprop	N-benzoyl-N-(3,4-dichlorophenyl)-DL-alanine
	bifenox	methyl 5-(2,4-dichlorophenoxy)-2-nitrobenzoat
35	bromacil	5-bromo-6-methyl-3-(1-methylpropyl)-2,4(1H,3H)pyrimidinedione

	<u>Common Name</u>	<u>Chemical Name</u>
	bromoxynil	3,5-dibromo-4-hydroxybenzonitrile
5	butachlor	N-(butoxymethyl)-2-chloro-N-(2,6-diethylphenyl)acetamide
	buthidazole	3-[5-(1,1-dimethylethyl)-1,3,4-thiadiazol-2-yl]-4-hydroxy-1-methyl-2-imidazolidinone
10	butralin	4-(1,1-dimethylethyl)-N-(1-methylpropyl)-2,6-dinitrobenzenamine
	butylate	S-ethyl bis(2-methylpropyl)carbamothioate
	cacodylic acid	dimethyl arsinic oxide
15	CDAA	2-chloro-N,N-di-2-propenylacetamide
	CDEC	2-chloroallyl diethyldithiocarbamate
	chloramben	3-amino-2,5-dichlorobenzoic acid
	chlorbromuron	3-(4-bromo-3-chlorophenyl)-1-methoxy-1-methylurea
20	chlorimuron ethyl	2-[[[(4-chloro-6-methoxy-2-pyrimidinyl)-amino]carbonyl]amino]sulfonyl]benzoic acid, ethyl ester
	chloroxuron	N'-[4-(4-chlorophenoxy)phenyl]-N,N-dimethylurea
25	chlorpropham	1-methylethyl 3-chlorophenylcarbamate
	chlorsulfuron	2-chloro-N-[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)amino]carbonyl]benzenesulfonamide
30	chlortoluron	N'-[3-chloro-4-methylphenyl]-N,N-dimethylurea
	cinmethylin	exo-1-methyl-4-(1-methylethyl)-2-[(2-methylphenyl)methoxy]-7-oxabicyclo[2.2.1]heptane
35	clethodim	(E,E)-(1)-2-[1-[[[(3-chloro-2-propenyl)-oxy]imino]propyl]-5-[2-(ethylthio)propyl]-3-hydroxy-2-cyclohexen-1-yl]

	<u>Common Name</u>	<u>Chemical Name</u>
5	clomazone	2-[(2-chlorophenyl)methyl]-4,4-dimethyl-3-isoxazolidinone
	cloproxydim	(E,E)-2-[1-[[[(3-chloro-2-propenyl)oxy]-imino]butyl]-5-[2-(ethylthio)propyl]-3-hydroxy-2-cyclohexen-1-one
	clopyralid	3,6-dichloro-2-pyridinecarboxylic acid
10	CMA	calcium salt of MAA
	cyanazine	2-[[[4-chloro-6-(ethylamino)-1,3,5-triazin-2-yl]amino]-2-methylpropanenitrile
	cycloate	S-ethyl cyclohexylethylcarbamothioate
15	cycluron	3-cyclooctyl-1,1-dimethylurea
	cyperquat	1-methyl-4-phenylpyridinium
	cyprazine	2-chloro-4-(cyclopropylamino)-6-(isopropylamino)-s-triazine
20	cyprazole	N-[5-(2-chloro-1,1-dimethylethyl)-1,3,4-thiadiazol-2-yl]cyclopropanecarboxamide
	cypromid	3',4'-dichlorocyclopropanecarboxanilide
	dalapon	2,2-dichloropropanoic acid
25	dazomet	tetrahydro-3,5-dimethyl-2H-1,3,5-thiadiazine-2-thione
	DCPA	dimethyl 2,3,5,6-tetrachloro-1,4-benzenedicarboxylate
	desmediphan	ethyl [3-[[[(phenylamino)carbonyl]oxy]-phenyl]carbamate
30	desmetryn	2-(isopropylamino)-4-(methylamino)-6-(methylthio)-s-triazine
	diallate	S-(2,3-dichloro-2-propenyl)bis(1-methylethyl)carbamothioate
35	dicamba	3,6-dichloro-2-methoxybenzoic acid
	dichlobenil	2,6-dichlorobenzonitrile

17

<u>Common Name</u>	<u>Chemical Name</u>
5 dichlorprop	(±)-2-(2,4-dichlorophenoxy)propanoic acid
dichlofop-methyl	(±)-2-[4-(2,4-dichlorophenoxy)phenoxy]-propanoic acid, methyl ester
diethatyl	N-(chloroacetyl)-N-(2,6-diethylphenyl)-glycine
10 difenzoquat	1,2-dimethyl-3,5-diphenyl-1H-pyrazolium
dinitramine	N ³ ,N ³ -diethyl-2,4-dinitro-6-(trifluoromethyl)-1,3-benzenediamine
dinoseb	2-(1-methylpropyl)-4,6-dinitrophenol
15 diphenamid	N,N-dimethyl-α-phenylbenzeneacetamide
dipropetryn	6-(ethylthio)-N,N'-bis(1-methylethyl)-1,3,5-triazine-2,4-diamine
diquat	6,7-dihydrodipyrido[1,2-a:2',1'-c]-pyrazinedium ion
20 diuron	N'-(3,4-dichlorophenyl)-N,N-dimethylurea
DNOC	2-methyl-4,6-dinitrophenol
DPX-M6316	3-[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)amino]carbonyl]amino]sulfonyl]-2-thiophenecarboxylic acid, methyl ester
25 DSMA	disodium salt of MAA
endothall	7-oxabicyclo[2.2.1]heptane-2,3-dicarboxylic acid
EPTC	S-ethyl dipropylcarbamothioate
30 ethalfluralin	N-ethyl-N-(2-methyl-2-propenyl)-2,6-dinitro-4-(trifluoromethyl)-benzenamine

35

	<u>Common Name</u>	<u>Chemical Name</u>
	ethofumesate	(±)-2-ethoxy-2,3-dihydro-3,3-dimethyl-5-benzofuranyl methanesulfonate
5	Express®	2-[[[N-(4-methoxy-6-methyl-1,3,5-triazine-2-yl)-N-methylamino]carbonylamino]-sulfonyl]benzoic acid, methyl ester
	fenac	2,3,6-trichlorobenzeneacetic acid
10	fenoxaprop	(±)-2-[4-[(6-chloro-2-benzoxazolyl)oxy]-phenoxy]propanoic acid
	fenuron	N,N-dimethyl-N'-phenylurea
	fenuron TCA	Salt of fenuron and TCA
15	flamprop	N-benzoyl-N-(3-chloro-4-fluorophenyl)-DL-alanine
	fluazifop	(±)-2-[4-[[5-(trifluoromethyl)-2-pyridinyl]oxy]phenoxy]propanoic acid
20	fluazifop-P	(R)-2-[4-[[5-(trifluoromethyl)-2-pyridinyl]oxy]phenoxy]propanoic acid
	fluchloralin	N-(2-chloroethyl)-2,6-dinitro-N-propyl-4-(trifluoromethyl)benzenamine
	fluometuron	N,N-dimethyl-N'-[3-(trifluoromethyl)-phenyl]urea
25	fluorochloridone	3-chloro-4-(chloromethyl)-1-[3-(trifluoromethyl)phenyl]-2-pyrrolidinone
	fluorodifen	p-nitrophenyl α,α,α-trifluoro-2-nitro-p-tolyl ether
30	fluoroglycofen	carboxymethyl 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitrobenzoate
	fluridone	1-methyl-3-phenyl-5-[3-(trifluoromethyl)phenyl]-4(1H)-pyridinone
	fomesafen	5-[2-chloro-4-(trifluoromethyl)phenoxy]-N-(methylsulfonyl)-2-nitrobenzamide
35		

	<u>Common Name</u>	<u>Chemical Name</u>
5	fosamine	ethyl hydrogen (aminocarbonyl)-phosphate
	glyphosate	N-(phosphonomethyl)glycine
	haloxyfop	2-[4-[[3-chloro-5-(trifluoromethyl)-2-pyridinyl]oxy]phenoxy]propanoic acid
10	hexaflurate	potassium hexafluoroarsenate
	hexazinone	3-cyclohexyl-6-(dimethylamino)-1-methyl-1,3,5-triazine-2,4(1H,3H)-dione
	imazamethabenz	6-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-m-toluic acid, methyl ester and 6-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-p-toluic acid, methyl ester
15	imazapyr	(±)-2-[4,5-dihydro-4-methyl-4-(1-methylethyl)-5-oxo-1H-imidazol-2-yl]-3-pyridinecarboxylic acid
20	imazaquin	2-[4,5-dihydro-4-methyl-4-(1-methylethyl)-5-oxo-1H-imidazol-2-yl]-3-quinolinecarboxylic acid
	imazethapyr	(±)-2-[4,5-dihydro-4-methyl-4-(1-methylethyl)-5-oxo-1H-imidazol-2-yl]-5-ethyl-3-pyridinecarboxylic acid
25	ioxynil	4-hydroxy-3,5-diiodobenzonitrile
	isopropalin	4-(1-methylethyl)-2,6-dinitro-N,N-dipropylbenzenamine
	isoproturon	N-(4-isopropylphenyl)-N',N'-dimethylurea
30	isouron	N'-(5-(1,1-dimethylethyl)-3-isoxazolyl)-N,N-dimethylurea
	isoxaben	N-[3-(1-ethyl-1-methylpropyl)-5-isoxazolyl]-2,6-dimethoxybenzamide
	karbutilate	3-[[[(dimethylamino)carbonyl]amino]-phenyl-(1,1-dimethylethyl)carbamate

	<u>Common Name</u>	<u>Chemical Name</u>
5	lactofen	(±)-2-ethoxy-1-methyl-2-oxoethyl 5-[(4-chloro-4-(trifluoromethyl)phenoxy)-2-nitrobenzoate
	lenacil	3-cyclohexyl-6,7-dihydro-1H-cyclopenta-pyrimidine-2,4(3H,5H)-dione
10	linuron	N'-(3,4-dichlorophenyl)-N-methoxy-N-methylurea
	MAA	methylarsonic acid
	MAMA	monoammonium salt of MAA
	MCPA	(4-chloro-2-methylphenoxy)acetic acid
15	MCPB	4-(4-chloro-2-methylphenoxy)butanoic acid
	mecoprop	(±)-2-(4-chloro-2-methylphenoxy)-propanoic acid
20	mefluidide	N-[2,4-dimethyl-5-[[[(trifluoromethyl)sulfonyl]amino]phenyl]acetamide
	methal-propalin	N-(2-methyl-2-propenyl)-2,6-dinitro-N-propyl-4-(trifluoromethyl)benzenamide
	methabenz-thiazuron	1,3-dimethyl-3-(2-benzothiazolyl)urea
25	metham	methylcarbamodithioic acid
	methazole	2-(3,4-dichlorophenyl)-4-methyl-1,2,4-oxadiazolidine-3,5-dione
	methoxuron	N'-(3-chloro-4-methoxyphenyl)-N,N-dimethylurea
30	metolachlor	2-chloro-N-(2-ethyl-6-methylphenyl)-N-(2-methoxy-1-methylethyl)acetamide

	<u>Common Name</u>	<u>Chemical Name</u>
	metribuzin	4-amino-6-(1,1-dimethylethyl)-3-(methy... thio)-1,2,4-triazin-5(4H)-one
5	metsulfuron methyl	2-[[[(4-methoxy-6-methyl-1,3,5-tri- azin-2-yl)amino]carbonyl]- amino]sulfonyl]benzoic acid, methyl ester
	MH	1,2-dihydro-3,6-pyridazinedione
10	molinate	S-ethyl hexahydro-1H-azepine-i-carbo- thioate
	monolinuron	3-(p-chlorophenyl)-1-methoxy-1-methyl- urea
	monuron	N'-(4-chlorophenyl)-N,N-dimethylurea
15	monuron TCA	Salt of monuron and TCA
	MSMA	monosodium salt of MAA
	napropamide	N,N-diethyl-2-(1-naphthalenylloxy)- propanamide
20	naptalam	2-[(1-naphthalenylamino)carbonyl]- benzoic acid
	neburon	1-butyl-3-(3,4-dichlorophenyl)-1-methyl- urea
25	nitralin	4-(methylsulfonyl)-2,6-dinitro-N,N- dipropylaniline
	nitrofen	2,4-dichloro-1-(4-nitrophenoxy)benzene
	nitrofluorfen	2-chloro-1-(4-nitrophenoxy)-4-(tri- fluoromethyl)benzene
30	norea	N,N-dimethyl-N'-(octahydro-4,7-methano- 1H-inden-5-yl)urea 3α, - 4α, 5α, 7α, 7αα-isomer
	norflurazon	4-chloro-5-(methylamino)-2-[3-(trifluoro- methyl)phenyl]-3(2H)-pyridazinone
35	oryzalin	4-(dipr pylamino)-3,5-dinitr - benzenesulf namide

	<u>Common Name</u>	<u>Chemical Name</u>
5	oxadiazon	3-[2,4-dichloro-5-(1-methylethoxy)-phenyl]-5-(1,1-dimethylethyl)-1,3,4-oxadiazol-2(3H)-one
	oxyfluorfen	2-chloro-1-(3-ethoxy-4-nitrophenoxy)-4-(trifluoromethyl)benzene
	paraquat	1,1'-dimethyl-4,4'-dipyridinium ion
10	pebulate	S-propyl butylethylcarbamothioate
	pendimethalin	N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitrobenzenamine
	perfluidone	1,1,1-trifluoro-N-[2-methyl-4-(phenylsulfonyl)phenyl]methanesulfonamide
15	phenmedipham	3-[(methoxycarbonyl)amino]phenyl (3-methylphenyl)carbamate
	picloram	4-amino-3,5,6-trichloro-2-pyridine-carboxylic acid
20	PPG-1013	5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitroacetophenone oxime-O-acetic acid, methyl ester
	procyazine	2-[[4-chloro-6-(cyclopropylamino)-1,3,5-triazine-2-yl]amino]-2-methylpropane-nitrile
25	profluralin	N-(cyclopropylmethyl)-2,6-dinitro-N-propyl-4-(trifluoromethyl)benzenamine
	prometon	6-methoxy-N,N'-bis(1-methylethyl)-1,3,5-triazine-2,4-diamine
	prometryn	N,N'-bis(1-methylethyl)-6-(methylthio)-1,3,5-triazine-2,4-diamine
30	pronamide	3,5-dichloro-N-(1,1-dimethyl-2-propynyl)benzamide
	propachlor	2-chloro-N-(1-methylethyl)-N-phenylacetamide
35	pr panil	N-(3,4-dichlorophenyl)pr panamid
	pr pazine	6-chloro-N,N'-bis(1-methylethyl)-1,3,5-triazine-2,4-diamine

	<u>Common Name</u>	<u>Chemical Name</u>
	propham	1-methylethyl phenylcarbamate
5	prosulfalin	N-[[4-(dipropylamino)-3,5-dinitro-phenyl]sulfonyl]-S,S-dimethylsulfilimine
	prynachlor	2-chloro-N-(1-methyl-2-propynyl)acetanilide
10	pyrazon	5-amino-4-chloro-2-phenyl-3(2H)-pyridazinone
	quizalofop ethyl	(±)-2-[4-[(6-chloro-2-quinoxalinyloxy]phenoxy]propanoic acid, ethyl ester
15	secbumeton	N-ethyl-6-methoxy-N'-(1-methylpropyl)-1,3,5-triazine-2,4-diamine
	sethoxydim	2-[1-(ethoxyimino)butyl]-5-[2-(ethylthio)propyl]-3-hydroxy-2-cyclohexen-1-one
20	siduron	N-(2-methylcyclohexyl)-N'-phenylurea
	simazine	6-chloro-N,N'-diethyl-1,3,5-triazine-2,4-diamine
	sulfometuron methyl	2-[[[(4,6-dimethyl-2-pyrimidinyl)amino]carbonylamino]sulfonyl]-benzoic acid, methyl ester
25	TCA	trichloroacetic acid
	tebuthiuron	N-[5-(1,1-dimethylethyl)-1,3,4-thiadiazol-2-yl]-N,N'-dimethylurea
	terbacil	5-chloro-3-(1,1-dimethylethyl)-6-methyl-2,4(1H,3H)-pyrimidinedione
30	terbuchlor	N-(butoxymethyl)-2-chloro-N-[2-(1,1-dimethylethyl)-6-methylphenyl]-acetamide

	<u>Common Name</u>	<u>Chemical Name</u>
5		
	terbuthyl- azine	2-(tert -butylamino)-4-chloro-6-(ethyl- amino)- s -triazine
	terbutol	2,6-di- tert -butyl-p-tolyl methylcar- bamate
10	terbutryn	N-(1,1-dimethylethyl)-N'-ethyl-6- (methylthio)-1,3,5-triazine- 2,4-diamine
	thiobencarb	S-[(4-chlorophenyl)methyl] diethylcar- bamothioate
15	triallate	S-(2,3,3-trichloro-2-propenyl) bis(1- methylethyl)carbamothioate
	triclopyr	[(3,5,6-trichloro-2-pyridinyl)- oxy]acetic acid
	tridiphane	2-(3,5-dichlorophenyl)-2-(2,2,2- trichloroethyl)oxirane
20	trifluralin	2,6-dinitro-N,N-dipropyl-4-(tri- fluoromethyl)benzenamine
	trimeturon	1-(p-chlorophenyl)-2,3,3-trimethylpseu- dourea
25	2,4-D	(2,4-dichlorophenoxy)acetic acid
	2,4-DB	4-(2,4-dichlorophenoxy)butanoic acid
	vernolate	S-propyl dipropylcarbamothioate
	xylachlor	2-chloro-N-(2,3-dimethylphenyl)-N- (1-methylethyl)acetamide
30		

The herbicidal properties of the subject com-
pounds were discovered in a number of greenhouse tests.
The test procedures and results follow.

Utility

The compound of the present invention is an active herbicide for selective and/or general broad-
5 leaf and grass weed control in non-crop situations, fallow, plantation crops such as coffee, tea, cocoa, oil palm, rubber, sugarcane, grapes, fruit trees, nut trees, banana, plantain, pineapple, citrus, cork, and certain conifers, and pasture and turf grasses such
10 as Kentucky bluegrass, fescue, annual and perennial ryegrass, bermudagrass, bentgrass, and bahiagrass.

Compound I can be applied as a preemergence or postemergence treatment using techniques of banding, directed sprays or broadcast applications. By
15 selecting the appropriate rate, the compound of this invention can be used in areas where complete control of all vegetation is desired, such as around fuel storage tanks, industrial storage areas, oil well sites, drive-in theatres, around billboards,
20 highways, utilities, and railroad structures. Further, the compound's rapid soil dissipation allows it to be used in fallow fields where the ability to replant sensitive crops is desired. Alternatively, by selecting the proper rates and adjuvants, the
25 compound of this invention can be used for selective control of weeds in plantation crops such as coffee, tea, cocoa, oil palm, rubber, grapes, fruit trees, nut trees, banana, plantain, pineapple, citrus, cork, and sugarcane, certain conifers, and in turf and
30 pasture grasses. In general, the compound of this invention is used at 0.5 to 500 g/ha with a preferred rate range of 1 to 250 g/ha. One skilled in the art can select the proper rate for a given situation.

5 The compound of this invention may be used in
combination with other herbicides. It is
particularly useful in combination with herbicides
used in fallow, total vegetation control, plantation
10 crops, certain conifers, pastures, and turfs. These
herbicides include triazine, triazole, uracil, urea,
amide, carbamate, bipyridylum, phenoxy, sulfonyl-
urea, and imidazolinone types of chemistries. It may
also be used in combination with mefluidide or
glyphosate.

15 More preferred mixtures for use on fallow land
would include DPX-M6316, Express®, difenzoquat,
diclofop-methyl, paraquat, glyphosate and
imazamethabenz.

20 The herbicidal properties of the subject
compound were discovered in a number of greenhouse
tests conducted as described below. Test results
clearly demonstrate the various herbicidal uses of
Compound I.

25 Test 1

Seeds of Bromus tectorum, Kochia scoparia,
Salsoa kali, Avena fatua, Convolvulus arvensis,
Secale cereale, Setaria viridis and Triticum aestivum
were placed in 26-cm plastic pans containing a
pasteurized sandy loam soil (pH 6.5, 1% organic
30 matter). Plantings were maintained in the greenhouse
for 18-28 days at which time the postemergence
treatments were applied using a nonphytotoxic solvent
as the carrier for the herbicide. The preemergence
segment of the test was seeded immediately before
herbicide application using Bromus tectorum, Kochia
35 scoparia, Polygonum convolvulus, Salsoa kali, Avena
fatua, Convolvulus arvensis, Secale cereale, Setaria

5 viridis, Aegilops cylindrica, Amaranthus retroflexus,
 Chenopodium album, Hordeum vulgare, Triticum
 aestivum, Sorghum bicolor, and Zea mays. All
 treatments were maintained in the greenhouse for an
 additional 21 days at which time visual assessment of
10 weed control was determined using a scale of 0 to 100
 for each species where 0 represented no control and
 100 represented complete control. Test results are
 shown in Table 1.

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Table 1

		<u>Rate of Active Ingredient (g/ha)</u>					
5	<u>Species</u>	<u>125</u>	<u>64</u>	<u>32</u>	<u>16</u>	<u>8</u>	<u>4</u>
	<u>POSTEMERGENCE</u>						
	<u>Kochia scoparia</u>	100	100	100	100	100	100
	<u>Salsoa kali</u>	100	100	100	100	100	100
	<u>Convolvulus arvensis</u>	100	100	100	100	100	90
10	<u>Avena fatua</u>	100	100	100	100	80	80
	<u>Bromus tectorum</u>	100	100	100	100	100	100
	<u>Secale cereale</u>	100	100	100	100	100	70
	<u>Setaria viridis</u>	100	100	100	100	90	90
	<u>Triticum aestivum</u>	100	100	100	100	100	90
15	<u>PREEMERGENCE</u>						
	<u>Amaranthus retroflexus</u>	100	100	100	90	90	70
	<u>Chenopodium album</u>	100	100	100	90	90	60
	<u>Convolvulus arvensis</u>	100	100	90	90	40	20
	<u>Kochia scoparia</u>	100	100	100	100	80	80
	<u>Polygonum convolvulus</u>	100	80	80	80	20	20
	<u>Salsoa kali</u>	100	100	80	70	70	50
20	<u>Aegilops cylindrica</u>	100	100	80	60	50	30
	<u>Avena fatua</u>	100	90	60	50	40	0
	<u>Bromus tectorum</u>	100	90	90	80	70	50
	<u>Hordeum vulgare</u>	90	80	80	70	60	50
	<u>Secale cereale</u>	90	80	70	40	30	20
	<u>Setaria viridis</u>	100	90	70	50	30	20
	<u>Sorghum bicolor</u>	100	100	100	100	100	90
25	<u>Triticum aestivum</u>	100	100	100	100	100	70
	<u>Zea mays</u>	100	90	90	90	50	40

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Test 2

Rhizomes of Agropyron repens and Cirsium arvense were planted in 17.5-cm plastic pots and grown in the greenhouse for 5 months prior to herbicide application. Plants were 24 to 35 cm tall and releasing lateral buds at the time of treatment. Compound I was diluted in water containing 0.25% v/v non-ionic surfactant and applied to the plants. The degree of weed control was visually rated 36 days after application of the herbicide. Control ratings were based upon comparison to an untreated control using a scale of 0 to 100 where 0 indicated no effect and 100 represented death of all foliar portions with no regrowth apparent. Results are given in Table 2.

Table 2

<u>Treatment</u>	<u>Rate g/ha</u>	<u>Agropyron repens</u>	<u>Cirsium arvense</u>
Compound I	64	95	50
	32	90	50
	16	85	40

Control ratings 36 DAT

Test 3

Seeds of crabgrass (*Digitaria* spp), guinea grass (*Panicum maximum*) and narrowleaf panicum (*P. maximum*) were planted in 15-cm plastic pots. The weeds were treated postemergence with compound I, formulated in a non-phytotoxic solvent, when the plants had 3 or more leaves and had tillered. The degree of weed control was visually rated 17 days after compound application (DAT). Control ratings were based on a scale of 0 to 100 where 0 = no effect, 20 = minimal control and 100 = complete control. Results are given in Table 3.

Table 3

<u>Control Rating 17 DAT</u>				
<u>Treatment</u>	<u>Rate g/ha</u>	<u>Crabgrass</u>	<u>Guinea Grass</u>	<u>Narrowleaf panicum</u>
Compound I	32	100	70	90
	16	70	-	-
	8	50	-	-
Check	0	0	0	0

- = not determined.

Test 4

The object of this test was to evaluate the efficacy of the compound of this invention on weeds that infest some plantation crops at different growth stages of the weeds. Asystasia intrusa, Mikania cordata, goldenrod Solidago spp, Boston fern Nephrolepis exalata, and lalang Imparata cylindrica were planted in 15-cm plastic pots. Plants were sprayed postemergence with Compound I in a non-phytotoxic solvent. The Asystasia plants ranged in growth from 4 leaves to 9 leaves and very branched, Mikania had 6 leaves to vining, the goldenrod was branching and the Boston fern was growing vigorously. The degree of weed control was visually rated 20 to 24 days after treatment (DAT). Control ratings are based on the scale of 0 to 100 where 0 = no effect, 20 = minimal control and 100 = complete control.

Variations in the results of the tests could be due to the fact that the tests were run at different times of the year or due to the stage of growth of the weeds. Test results are given in Tables 4 to 8.

Table 4Control Rating 24 DAT

<u>Treatment</u>	<u>Rate g/ha</u>	<u>Asystasia intrusa*</u>	<u>Nephrolepis exalata</u>
Compound I	64	60	80
	32	50	60
	16	40	40
	8	30	0
	4	20	0
	0	0	0
Check			

*At 4-leaf stage

32

Table 5

Control Rating 20 DAT

5	Treatment	Rate g/ha	Mikania ¹ cordata	Asystasia ² intrusa
	Compound I	64	30	100
		32	0	90
		16	0	60
		8	0	0
	Check	0	0	0
10	<hr/>			

¹ At 6 to 8-leaf stage² At 6 to 9-leaf stage

Table 6

Control Rating 20 DAT

15	Treatment	Rate g/ha	Mikania* cordata	Asystasia* intrusa	Goldenrod*
	Compound I	64	0	30	-
		32	0	0	30
		16	0	0	30
20		8	0	0	-
	Check	0	0	0	0
	<hr/>				

- = not determined

*Profusely branching

25

Table 7

Control Rating 22 DAT

	Treatment	Rate g/ha	Mikania* cordata
30	Compound I	64	20
		32	0
		16	0
		8	0
	Check	0	0

*Vining and profusely branching.

35

Table 8
Control Rating 22 DAT

<u>Treatment</u>	<u>Rate</u> <u>g/ha</u>	<u>Mikania*</u> <u>cordata</u>	<u>Lalang</u>
Compound I	64	20	40
	32	0	20
	16	0	20
Check	0	0	0

Test 5

Sugarcane (Saccharum spp.) cuttings which had been pre-sprouted were planted in 15-cm plastic pots. Narrowleaf panicum (Panicum maximum), large crabgrass (Digitaria sanguinalis), guineagrass (P. maximum) and itchgrass (Rottboellia exaltata) were seeded in individual 15-cm plastic pots.

The pots were treated postemergence when the plants had 3 to 4 leaves with 2 to 3 tillers with Compound I formulated in a non-phytotoxic carrier made up of acetone-humectant-water-surfactant (22:1:1:0.05 v/v). The degree of crop injury and weed control was visually rated 27 days after treatment (DAT). Plant injury ratings are based on the scale of 0 to 100 where 0 = no effect, 20 = minimal injury and 100 = complete control. Test results are shown in Table 9.

Table 9Plant Injury Rating 27 DAT

<u>Treatment</u>	<u>Rate</u> <u>g/ha</u>	<u>Sugar-</u> <u>cane</u>	<u>Narrow-</u> <u>leaf</u> <u>panicum</u>	<u>Large</u> <u>Crab-</u> <u>grass</u>	<u>Guinea</u> <u>Grass</u>	<u>Itch-</u> <u>grass</u>
Compound I	64	30	100	100	100	100
	32	20	100	80	80	100
	16	20	100	60	70	80
	8	10	70	30	30	80
Check	0	0	0	0	0	0

Test 6

The object of this test was to evaluate the effect of surfactants and plant growth stage on the efficacy of the compound of this invention. Plastic windowsill pots were filled with planting medium and seeded with yellow nutsedge (Cyperus esculentus), large crabgrass (Digitaria sanguinalis), goosegrass (Elsusine indica), bermudagrass (Cynodon dactylon), broadleaf signalgrass (Brachiara platyphalla), smooth crabgrass (Digitaria ischaemum), guinea grass (Panicum maximum) and johnsongrass (Sorghum halapense). Coffee (Coffea spp) plants with 2 to 4 branches were included in one test.

Plants were treated postemergence with Compound I formulated with different surfactants or a non-phytotoxic carrier made up of acetone-humectant-water-surfactant. The results showed that addition of a surfactant could improve the weed control, while maintaining safety to coffee.

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Test 7

Individual 11.4-cm plastic pots filled with planting medium were seeded with rape, wild radish (Raphanus raphanistrum), tansy mustard (Descurainia pinnata), ball mustard (Neslia paniculata), black mustard (Brassica nigra), yellow mustard, brown mustard, lentils, safflower, flax, sunflower, peanut, vetch, alfalfa, field bean, pea, violet (Viola spp), Kochia (Kochia spp), Russian thistle (Salsola spp), Matricaria spp, groundsel (Senecio spp), shepherds-purse (Capsella bursa-pastoris), nightshade (Solanum spp), sowthistle (Sonchus spp), sugar beet, wheat, "Bonanza" barley and "Morex" barley, Kentucky bluegrass (Poa pratensis), red clover (Trifolium pratense), dandelion (Taraxacum spp) and buckhorn plantain (Plantago lanceolata).

Plants were treated postemergence with the compound formulated with a non-phytotoxic carrier. Plants were visually rated 18 days after treatment (DAT). Response ratings were based on the scale of 0 to 100 where 0 = no effect, 20 = minimal injury and 100 = complete control. Test results are shown in Table 10.

36

Table 10

Plant Injury Rating 18 DAT
Compound I Check

5	<u>Rate (g/ha)</u>	<u>32</u>	<u>16</u>	<u>0</u>
	Rape	100	80	0
	Wild radish	100	100	0
	Wild mustard	100	100	0
	Tansy mustard	100	100	0
	Ball mustard	100	100	0
	Black mustard	100	100	0
10	Yellow mustard	100	100	0
	Brown mustard	100	100	0
	Onicuta mustard	100	100	0
	Lentils	100	100	0
	Safflower	100	100	0
	Flax	100	100	0
	Sunflower	100	100	0
	Peanut	100	100	0
15	Vetch	100	100	0
	Alfalfa	100	100	0
	Field bean	100	100	0
	Pea	100	100	0
	Viola	100	100	0
	Kochia	100	100	0
	Russian thistle	100	100	0
20	Matricaria	100	100	0
	Groundsel	100	100	0
	Sheperdspurse	100	100	0
	Nightshade	60	50	0
	Sowthistle	100	100	0
	Sugar beet	100	100	0
	Wheat	100	100	0
	Bonanza barley	100	90	0
25	Morex barley	100	90	0
	Kentucky bluegrass	100	100	0
	Red clover	100	100	0
	Dandelion	100	100	0
	Buckhorn plantain	50	30	0

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Test 8

The object of this test was to evaluate the compound of this invention for postemergence safety to pineapple, turfs and weed control. Climax timothy, Kentucky bluegrass, forage fescue, Kentucky fescue, bermudagrass, bentgrass, annual bluegrass, bahiagrass, purple nutsedge, yellow nutsedge, large crabgrass, perennial ryegrass, annual ryegrass, galium and pineapple were sprayed postemergence with the compound in a non-phytotoxic solvent. Plants were visually rated 36 days after treatment (DAT). Injury ratings were based on the scale of 0 to 100 where 0 = no effect, 20 = minimal effect and 100 = complete control. Test results are shown in Table 11.

Table 11

		<u>Plant Injury Rating 36 DAT</u>		
		<u>Compound I</u>		<u>Check</u>
20	<u>Rate (g/ha)</u>	<u>32</u>	<u>16</u>	<u>0</u>
	Climax timothy	100	100	0
	Kentucky bluegrass	100	100	0
	Forage fescue	100	100	0
	Galium	100	100	0
	Pineapple	0	0	0
	Kentucky fescue	100	100	0
25	Bermudagrass	0	0	0
	Bentgrass	100	70	0
	Annual bluegrass	100	100	0
	Bahiagrass	100	70	0
	Purple nutsedge	100	50	0
	Yellow nutsedge	100	60	0
	Large crabgrass	80	60	0
30	Perennial ryegrass	100	100	0
	Annual ryegrass	100	100	0

Test 9

Plastic windowsill pots containing planting medium were seeded with bahiagrass, bindweed (*Convolvulus* spp), guinea grass (*Panicum maximum*), large crabgrass (*Digitaria sanguinalis*) and pigweed (*Amaranthus* spp). Individual 15-cm plastic pots contained sandbur (*Cenchrus echinatus*), *Mikania cordata*, Boston fern (*Nephrolepis exalata*), quackgrass (*Agropyron repens*), elephantgrass (*Pennisetum purpureum*) and sowthistle (*Sonchus* spp) plants.

Plants were treated postemergence with Compound I formulated in a non-phytotoxic solvent. Plants were visually rated 22 days after treatment (DAT). Injury and weed control ratings were based on the scale of 0 to 100 where 0 = no effect, 20 = minimal control and 100 = complete control. Test results are shown in Table 12.

Table 12Plant Injury Rating 22 DAT

	<u>Compound I</u>			<u>Check</u>
<u>Rate (g/ha)</u>	<u>32</u>	<u>16</u>	<u>8</u>	<u>0</u>
Sandbur	100	100	100	0
<i>Mikania cordata</i>	30	0	0	0
Boston fern	80	50	40	0
Sowthistle	100	70	60	0
Quackgrass	100	90	70	0
Lalang	20	0	0	0
Elephantgrass	70	50	0	0
Bahiagrass	-	40	-	0
Bindweed	-	70	-	0
Guinea grass	-	60	-	0
Large crabgrass	-	60	-	0
Pigweed	-	90	-	0
Coffee	0	0	0	0
Sugarcane	30	20	10	0

- = not determined

Test 10

The object of these tests was to evaluate the compound of this invention for postemergence safety to crops and weed control. Seedling and budded Macedine grapes were grown in 11.4-cm pots. Flax, green foxtail (Setaria viridis), "Morex" barley, wild oats (Avena fatua), "Era" wheat, annual ryegrass (Lolium multiflorum), wild radish (Raphanus raphanistrum), rape, sunflower, chickweed (Stellaria media) and wild buckwheat (Polygonum convolvulus) were seeded in windowsill pots.

The plants were sprayed postemergence with Compound I in a non-phytotoxic solvent. Plants were visually rated 15 and 27 days after treatment (DAT). Injury and weed control ratings were based on the scale of 0 to 100 where 0 = no effect, 20 = minimal control and 100 = complete control. Test results are shown in Tables 13 and 14.

Table 13Plant Injury Rating 27 DAT

<u>Treatment</u>	<u>Rate g/ha</u>	<u>Grapes</u>	
		<u>Seedling</u>	<u>Budded</u>
Compound I	32	20	10
	16	20	10
	8	10	0
	4	0	0
	2	0	0
Check	1	0	0
	0	0	0

Table 14

5	Plant Injury Rating 15 DAT		
	Compound I	Check	
	Rate (g/ha)	16	0
	Flax	100	0
	Green foxtail	100	0
10	Morex barley	100	0
	Wild oats	100	0
	Era wheat	100	0
	Annual ryegrass	100	0
	Wild radish	100	0
	Rape	100	0
	Sunflower	100	0
15	Chickweed	100	0
	Wild buckwheat	100	0

Test 11

Budded citrus seedlings were planted in
 30-liter plastic pots which were also seeded with
 sandbur, guineagrass, narrowleaf panicum, common
 ragweed, yellow nutsedge, purple nutsedge and
 bindweed. Compound I was applied preemergence and
 postemergence to the weeds. Treatments were sprayed
 to simulate the trunk-to-trunk herbicide application
 method used in some citrus groves. The compound was
 also applied postemergence to weeds grown in
 windowsill flats. The flats were filled with
 planting medium and seeded with morningglory (Ipomoea
spp.), purple nutsedge (Cyperus rotundus), pigweed
 (Amaranthus spp.), bindweed (Convolvulus spp.),
 narrowleaf panicum (Panicum maximum), common ragweed
 (Ambrosia artemisiifolia), sandbur (Cenchrus
echinatus), itchgrass (Rottboellia exaltata),
 johnsongrass (Sorghum halepense), guineagrass
 (Panicum maximum), smooth crabgrass (Digitaria
ischaemum), broadleaf signalgrass (Brachiaria

platyphylla), bermudagrass (Cynodon dactylon),
 goosegrass (Eleusine indica), large crabgrass
 (Digitaria sanguinalis) and yellow nutsedge (Cyperus
esculentus).

The compound was formulated and sprayed with
 0.25% X-77 surfactant in water. Plants were visually
 rated 21 and 29 days after treatment and compared
 with appropriate controls. Injury ratings were based
 on the scale of 0 to 100, where 0 indicates no
 effect, 20 indicates minimum effect and 100 indicates
 complete control. Variations in the results of this
 test with others could be due to the fact that the
 tests were conducted at different times of the year.
 Test results are shown in Tables 15 and 16.

Table 15

Compound I

Species	Preemergence	Postemergence
	32 g/ha	32 g/ha
-----	-----	-----
Citrus	0	0
Sandbur	100	100
Guineagrass	100	100
Narrowleaf panicum	80	90
Common ragweed	20	0
Yellow nutsedge	100	100
Purple nutsedge	100	90
Bindweed	90	100

Table 16

Species	Compound I
	Postemergence 32 g/ha
Morningglory	90
Purple nutsedge	100
Pigweed	100
Bindweed	100
Narrowleaf panicum	90
Common ragweed	0
Sandbur	100
Itchgrass	100
Johnsongrass	100
Guineagrass	100
Smooth crabgrass	100
Broadleaf signalgrass	100
Bermudagrass	20
Goosegrass	90
Large crabgrass	80
Yellow nutsedge	80

Test 12

The object of this test was to evaluate the safety of sour lemon to the over-the-top application of Compound I and control of lantana (Lantana camara) and trumpet creeper (Campsis radicans). Plants were sprayed postemergence with Compound I formulated in a non-phytotoxic solvent. Treated plants were visually rated 26 days after treatment and compared with appropriate controls. Injury ratings were based on the scale of 0 to 100, where 0 indicates no effect, 20 indicates minimal effect and 100 indicates complete control. Results are shown in Table 17.

Table 17Compound I

	<u>Species</u>	<u>Postemergence</u>		
		<u>32</u>	<u>16</u>	<u>8 g/ha</u>
5				
10	Sour Lemon	70	50	20
	Lantana	30	10	0
	Trumpet creeper	40	20	0

Test 13

15 Conifer species (Loblolly pine) seedlings were transplanted into 15-cm plastic pots filled with planting medium. Compound I was applied postemergence to the loblolly pine seedlings. The compound was formulated in a non-phytotoxic spray solvent. Plants were visually rated 240 days after treatment (DAT) and compared with appropriate controls. Injury ratings are based on the scale of 0 to 100, where 0 indicates no effect, 20 indicates minimal injury and 100 indicates complete kill. Results are shown in Table 18.

20

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Table 18Plant Injury Rating 240 DAT

	<u>Treatment</u>	<u>Rate (g/ha)</u>	<u>Loblolly pine</u>
	-----	-----	-----
30	Compound I	125	0
		64	0
		32	0
		16	0
		8	0
35	Check	0	0

CLAIMS

What is claimed is:

- 5 1. A method for controlling undesired weeds in noncrop areas, fallow land, turfs, pastures, certain conifers and plantation crops which comprises applying to the locus a herbicidally effective amount of 2-[[[(4,6-dimethoxypyrimidin-2-yl)aminocarbonyl]-
10 aminosulfonyl]-3-pyridinecarboxylic acid, methyl ester.
2. A method as in Claim 1, where the locus to be protected is a noncrop area.
3. A method as in Claim 1, where the locus
15 to be protected is fallow land.
4. A method as in Claim 3, wherein after the application, a crop is planted during the next growing season.
5. A method as in Claim 4 wherein the crop
20 is wheat or barley.
6. A method as in Claim 1 wherein the locus to be protected is a locus where turf and pasture grasses are grown.
7. A method as in Claim 1 wherein the locus
25 to be protected is where a plantation crop is grown.
8. A method as in Claim 7 wherein the plantation crop is coffee.
9. A method of Claim 7 wherein the plantation crop is tea.
- 30 10. A method as in Claim 7 wherein the plantation crop is cocoa.
11. A method as in Claim 7 wherein the plantation crop is rubber or oil palm.
12. A method as in Claim 7 wherein th
35 plantati n crop is sugarcane.

13. A method as in Claim 7 wher in th
plantation crop is banana.

14. A method as in Claim 7 wherein the
plantation crop is pineapple.

15. A method as in Claim 1 wherein the locus
to be protected is a conifer planting.

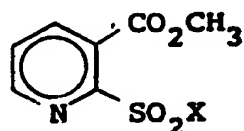
16. A method as in Claim 1 wherein the locus
to be protected is a grape planting.

17. A method as in Claim 1 wherein the locus
to be protected is a citrus planting.

18. A method as in Claim 1 wherein the locus
to be protected is a fruit tree planting.

19. A method as in Claim 1 wherein the locus
to be protected is a nut tree planting.

20. A 3-pyridinecarboxylic acid, methyl
ester, represented by the formula II:



II

wherein

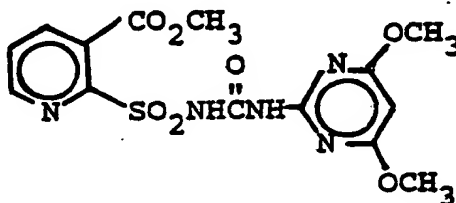
X is Cl, NH_2 , $\text{NHC}(\text{CH}_3)_3$ and $\text{HNC}(\text{O})\text{OC}_6\text{H}_5$.

21. The compound of Claim 20, 2-(aminosulfonyl)-
3-pyridinecarboxylic acid, methyl ester.



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁴ : A01N 47/36, C07D 213/80		(11) International Publication Number: WO 89/ 02700
A3		(43) International Publication Date: 6 April 1989 (06.04.89)
(21) International Application Number: PCT/US88/03276 (22) International Filing Date: 27 September 1988 (27.09.88) (31) Priority Application Numbers: 101,703 232,465 (32) Priority Dates: 28 September 1987 (28.09.87) 16 August 1988 (16.08.88) (33) Priority Country: US (60) Parent Application or Grant (63) Related by Continuation US 101,703 (CIP) Filed on 28 September 1987 (28.09.87) (71) Applicant (for all designated States except US): E.I. DU PONT DE NEMOURS AND COMPANY [US/US]; 1007 Market Street, Wilmington, DE 19898 (US). (72) Inventors; and (75) Inventors/Applicants (for US only) : LIANG, Paul, Hsiao-Tseng [US/US]; 3428 Pebble Beach Drive, Wilmington, DE 19808 (US). AMUTI, Kofi, Sam [GH/US]; 5412 Valley Green Drive, Wilmington, DE 19808 (US). RARDON, Patrick, Lee [US/US]; 191 Ash Lane, Elkton, MD 21921 (US).		(74) Agent: GREGORY, Theodore, C.; E.I. du Pont de Nemours and Company, 1007 Market Street, Wilmington, DE 19898 (US). (81) Designated States: AT (European patent), AU, BB, BE (European patent), BG, BJ (OAPI patent), BR, CF (OAPI patent), CG (OAPI patent), CH (European patent), CM (OAPI patent), DE (European patent), DK, FI, FR (European patent), GA (OAPI patent), GB (European patent), HU, IT (European patent), JP, KP, KR, LK, LU (European patent), MC, MG, ML (OAPI patent), MR (OAPI patent), MW, NL (European patent), NO, RO, SD, SE (European patent), SN (OAPI patent), SU, TD (OAPI patent), TG (OAPI patent), US. Published <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i> (88) Date of publication of the international search report: 20 April 1989 (20.04.89)

(54) Title: **HERBICIDAL o-CARBOMETHOXYLSULFONYLUREAS**

(I)

(57) Abstract

The compound of formula (I) is useful as an agricultural chemical. It has high herbicidal activity, desirable spectrum of weed control and low residual activity, as such, it is particularly useful for fallow.

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INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 88/03276

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ⁶ According to International Patent Classification (IPC) or to both National Classification and IPC IPC ⁴ : A 01 N 47/36; C 07 D 213/80											
II. FIELDS SEARCHED <div style="text-align: center; border-top: 1px solid black; border-bottom: 1px solid black;">Minimum Documentation Searched ⁷</div> <table style="width: 100%; border-collapse: collapse;"> <tr> <th style="width: 25%; text-align: left; border-bottom: 1px solid black;">Classification System</th> <th style="width: 75%; text-align: left; border-bottom: 1px solid black;">Classification Symbols</th> </tr> <tr> <td style="vertical-align: top; padding: 5px;">IPC⁴</td> <td style="vertical-align: top; padding: 5px;">A 01 N</td> </tr> </table> <div style="text-align: center; border-top: 1px solid black; border-bottom: 1px solid black;">Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁸</div>			Classification System	Classification Symbols	IPC ⁴	A 01 N					
Classification System	Classification Symbols										
IPC ⁴	A 01 N										
III. DOCUMENTS CONSIDERED TO BE RELEVANT ⁹ <table style="width: 100%; border-collapse: collapse;"> <tr> <th style="width: 10%; text-align: left; border-bottom: 1px solid black;">Category ⁹</th> <th style="width: 70%; text-align: left; border-bottom: 1px solid black;">Citation of Document ¹¹ with indication, where appropriate, of the relevant passages ¹²</th> <th style="width: 20%; text-align: left; border-bottom: 1px solid black;">Relevant to Claim No. ¹³</th> </tr> <tr> <td style="vertical-align: top; padding: 5px;">X</td> <td style="vertical-align: top; padding: 5px;"> EP, A, 0013480 (DU PONT) 23 July 1980 see page 24, line 24; claim 16 & US, A, 4435206 (cited in the application) -- </td> <td style="vertical-align: top; padding: 5px;">1-19</td> </tr> <tr> <td style="vertical-align: top; padding: 5px;">X</td> <td style="vertical-align: top; padding: 5px;"> EP, A, 0184385 (ISHIHARA SANGYO KAISHA) 11 June 1986 see page 19, no. 9 and page 23, tables 5,6 cited in the application ----- </td> <td style="vertical-align: top; padding: 5px;">1,6</td> </tr> </table>			Category ⁹	Citation of Document ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³	X	EP, A, 0013480 (DU PONT) 23 July 1980 see page 24, line 24; claim 16 & US, A, 4435206 (cited in the application) --	1-19	X	EP, A, 0184385 (ISHIHARA SANGYO KAISHA) 11 June 1986 see page 19, no. 9 and page 23, tables 5,6 cited in the application -----	1,6
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X	EP, A, 0184385 (ISHIHARA SANGYO KAISHA) 11 June 1986 see page 19, no. 9 and page 23, tables 5,6 cited in the application -----	1,6									
<div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p>¹⁰ Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 45%;"> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</p> <p>"G" document member of the same patent family</p> </div> </div>											
IV. CERTIFICATION <table style="width: 100%; border-collapse: collapse;"> <tr> <td style="width: 50%; border-bottom: 1px solid black; padding: 5px;">Date of the Actual Completion of the International Search</td> <td style="width: 50%; border-bottom: 1px solid black; padding: 5px;">Date of Mailing of this International Search Report</td> </tr> <tr> <td style="padding: 5px;">18th January 1989</td> <td style="padding: 5px;">28 MAR 1989</td> </tr> <tr> <td style="border-bottom: 1px solid black; padding: 5px;">International Searching Authority</td> <td style="border-bottom: 1px solid black; padding: 5px;">Signature of Authorized Officer</td> </tr> <tr> <td style="padding: 5px;">EUROPEAN PATENT OFFICE</td> <td style="padding: 5px;"> P.C.G. VAN DER PUTTEN </td> </tr> </table>			Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	18th January 1989	28 MAR 1989	International Searching Authority	Signature of Authorized Officer	EUROPEAN PATENT OFFICE	P.C.G. VAN DER PUTTEN	
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FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET

V. ☐ OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE ¹

This International search report has not been established in respect of certain claims under Article 17(2) (a) for the following reasons:

1. ☐ Claim numbers _____ because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claim numbers _____, because they relate to parts of the International application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claim numbers _____, because they are dependent claims and are not drafted in accordance with the second and third sentences of PCT Rule 6.4(a).

VI. ☒ OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING ²

This International Searching Authority found multiple inventions in this International application as follows:

Please see Form PCT/ISA/206 dated January 30th, 1989.

1. ☐ As all required additional search fees were timely paid by the applicant, this International search report covers all searchable claims of the International application.
2. ☐ As only some of the required additional search fees were timely paid by the applicant, this International search report covers only those claims of the International application for which fees were paid, specifically claims:
3. ☒ No required additional search fees were timely paid by the applicant. Consequently, this International search report is restricted to the invention first mentioned in the claims; it is covered by claim numbers:
1-19
4. ☐ As all searchable claims could be searched without effort justifying an additional fee, the International Searching Authority did not invite payment of any additional fee.

Remark on Protest

- ☐ The additional search fees were accompanied by applicant's protest.
☐ No protest accompanied the payment of additional search fees.

**ANNEX TO THE INTERNATIONAL SEARCH REPORT
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US 8803276
SA 25311

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on 20/03/89
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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP-A- 0013480	23-07-80	JP-A- 55102577	05-08-80
		EP-A- 0049554	14-04-82
		CA-A- 1114819	22-12-81
		CA-A- 1128047	20-07-82
		US-A- 4522645	11-06-85
		US-A- 4435206	06-03-84
		AU-A- 5338279	12-06-80
		AU-B- 526899	03-02-83
		US-A- 4544401	01-10-85
EP-A- 0184385	11-06-86	JP-A- 61267576	27-11-86
		US-A- 4744814	17-05-88

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